

VIA U.S. MAIL

January 19, 1995 95RD0308

Defense Technical Information Center Bldg. 5, Cameron Station Alexandria, Virginia 22304-6145

Subject:

Transmittal of Progress Report Entitled, "In-Situ Composites in the Aluminum Nitride-Alumina System," Under Contract No. N0014-94-C0263, Subline Item No. 0001AD (QUEST Proposal No. RD224028)

Dear Director:

The following is a summary of our activities on the subject contract between December 21, 1994 and January 19, 1995.

Task 3. Sintering and Microstructural Evaluation

The compositions in Table 1 were sintered as reported in the previous progress report. Sintered dimension were measured; shrinkages were calculated. Densities were measured by the liquid immersion technique. The results are tabulated in Table 2 where:

Wg = green weight

Wf = sintered weight

Ws = submersed weight

Dg = green diameter

Df = sintered diameter tg = green thickness

tf = sintered thickness

d = density

wl = weight loss

The results of X-ray diffraction studies are given in Table 3. As predicted by the AlN-Al₂O₃ phase diagram, the samples are composed of AlN and AlON polytype phases. However, the presence of three phases in two phase regions suggest that either the AlN- Al₂O₃ phase diagram of McCauley and Corbin is inaccurate or a one hour holding time was inadequate to reach phase equilibrium because of the possibly exothermic nature of the AlN- Al₂O₃ reactions. A complete understanding of this issue is not within the scope of the Phase I and should be pursued in a Phase

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Feb 1, 1995

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Code A. Watson

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- 2. The Defense Technical Information Center received the enclosed report (referenced below) which is not marked in accordance with the above reference.

Contract n00014-94-C0263
"In-Situ Composites in the aluminum Nitride-Alumina system,"

- 3. We request the appropriate distribution statement be assigned and the report returned to DTIC within 5 working days.
- 4. Approved distribution statements are listed on the reverse of this letter. If you have any questions regarding these statements, call DTIC's Cataloging Branch, (703) 274-6837.

FOR THE ADMINISTRATOR:

1 Encl

GOPALAKRISHNAN NAIR Chief, Cataloging Branch

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Defense Technical Information Center January 19, 1995 Page Two

II program. Preliminary microstructural studies showed the presence of fibrous microstructures in samples of compositions D and E. Further microstructural analysis is being carried out on samples having compositions C, D, E, and F.

Based on these results, we have chosen compositions D and E for doping with Y_2O_3 , and $Y_2O_3+SiO_2$. The powders are being spray dried.

Schedule

The program is two weeks behind schedule because of the two week holiday break at both QUEST and the University of Washington. As of January 1, 1995, 40% of the project budget had been spent.

Future Work

- 1. Finish microstructural analysis on samples of compositions C, D, E, and F.
- 2. Finish spray drying doped D and E compositions.
- 3. Press (5x0.6) cm disks of compositions D, E, and doped with D and E.
- 4. Sinter the disks and prepare mechanical test bars. Ten (10) test bars are planned from each compositions.
- 5. Prepare final report.

Should you have any questions or comments regarding this report or the program, please contact me at (206) 872-9500.

Sincerely,

Ender Savrun, Ph.D.

Senior Scientist

Research Division

ES:sh/95RD0308.W4W

cc: Contract Administration, (AINCOMP)

Accesion For

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Justification

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Table 1 Experimental Compositions

% wt			% mol		
Sample	AIN	Al_20_3	AIN	Al_20_3	
A	28.67	71.33	50	50	
В	32.94	67.06	55	45	
C	37.61	62.39	60	40	
D	42.74	57.26	65	35	
E	69.49	30.51	85	15	
F	88.42	11.58	95	5	
G	30.76	69.24	52.5	47.5	

Table 2 Experimental Results

Sample	Wg, g	Wf, g	Ws, g	Dg, mm	tg, mm	Df, mm	tf, mm	d, g/cm3	D, %	t, %	WL, %
•	RUN I	1900 C	1 HR								
A19	1.1036	1.095	0.8491	16.5354	2.38	14.019	2.053	3.524573	15.21826	13.7395	0.779268
A25	1.0942	1.083	0.8407	16.5354	2.39	14.039	2.002	3.53774	15.09731	16.23431	1.023579
B4	1.1015	1.093	0.8449	16.5616	2.403	14.021	2.06	3.486939	15.34031	14.27382	0.771675
B6	1.1044	1.097	0.8496	16.5636	2.3663	14.073	2.038	3.509602	15.03659	13.87398	0.670047
C3	1.1077	1.1	0.8484	16.5223	2.381	14.116	2.054	3.460453	14.56395	13.73373	0.695134
C4	1.1134	1.107	0.8541	16.5223	2.392	14.114	2.056	3.464573	14.57606	14.04682	0.574816
D4	1.1121	1.105	0.8479	16.56	2.412	14.168	2.071	3.401818	14.44444	14.13765	0.638432
D9	1.1175	1.11	0.8524	16.561	2.431	14.148	2.099	3.410578	14.57038	13.65693	0.671141
E1	1.1145	1.107	0.822	16.564	2.582	14.323	2.263	3.074353	13.52934	12.35476	0.672948
E6	1.1273	1.12	0.8315	16.561	2.623	14.314	2.281	3.072721	13.56802	13.03851	0.647565
F7	1.1116	1.108	0.8181	16.565	2.758	14.095	2.367	3.025119	14.91096	14.17694	0.323858
F9	1.1102	1.106	0.8139	16.564	2.763	14.144	2.385	2.996915	14.61	13.68078	0.37831
G5	1.1144	1.106	0.8547	16.555	2.367	14.122	2.043	3.483482	14.69647	13.68821	0.753769
G10	1.1209	1.113	0.8603	16.559	2.379	14.154	2.052	3.486108	14.52382	13.74527	0.704791
	RUN 2	2200 C	1 HR								
E3	1.1151	0.974	0.7251	16.564	2.598	14.077	2.28	3.097312	15.01449	12.24018	12.65357
F15	1.119	1.023	0.7421	16.564	2.771	14.285	2.434	2.882536	13.75875	12.16167	8.579088
	RUN 3	2100 C	1 HR								
E2	1.1242	1.1237	0.8331	16.564	2.616	14.259	2.292	3.060594	13.91572	12.38532	0.044476
1.2	RUN 4	2000 C	1 HR								
A14	1.105	1.0729	0.836	16.5354	2.41	13.808	2.077	3.584636	16.49431	13.81743	2.904977
B15	1.1032	1.0779	0.8384	16.558	2.372	13.944	2.024	3.562246	15.78693	14.67116	2.293328
C10	1.1159	1.093	0.8481	16.554	2.395	13.953	2.068	3.532501	15.71221	13.65344	2.052155
D12	1.1242	1.0963	0.8464	16.556	2.433	13.983	2.092	3.472275	15.54119	14.01562	2.481765
E7	1.1099	1.0861	0.7959	16.562	2.582	14.482	2.285	2.962261	12.55887	11.50271	2.144337
F3	1.1291	1.106	0.8024	16.566	2.797	14.309	2.428	2.883396	13.62429	13.19271	2.045877
G9	1.1173	1.085	0.8446	16.553	2.374	13.962	2.044	3.572286	15.65275	13.90059	2.890898
	RUN 5	1950 C	1 HR								
A17	1.0901	1.0765	0.8372	16.5354	2.38	13.906	2.05	3.560592	15.90164	13.86555	1.247592
B10	1.0802	1.0674	0.8299	16.552	2.334		2.005	3.557251	15.70203	14.09597	1.184966
C18	1.1156	1.1027	0.854	16.561	2.388	14.02	2.059	3.509397	15.34328		
D7	1.1237	1.11	0.8548	-	2.454	14.049	2.12	3.442653	15.16817	13.61043	
E11	1.1247	1.1085	0.812	15.564	2.615	14.563	2.322	2.959116	6.431509	11.20459	1.440384
F8	1.1132	1.0969	0.8017	16.565	2.769	14.333	2.414	2.941045	13.47419	12.82051	1.464247
G15	1.1271	1.1018	0.8565			13.958	2.055	3.555135	15.67692	13.65546	2.244699
	RUN 7	2050 C	1 HR								
E17	1.1196		0.8016	16.566	2,607	14.316	2.297	3.02475	13.58204	11.89106	3.027867
F14	1.1215		0.8002						14.03127	13.60029	
	RUN 6	2050 C *									
E4	1.1284		0.8156	16.565	2.622	14.302	2.287	3.042363	13.66133	12.77651	2.304147
F11	1.1245			16.563							

Table 3 Phase Compositions of Sintered Samples

	TEMPERATURE								
SAMPLE	1900°C	1950°C	2000°C	2050°C	2100°C	2200°C			
A	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) SE	M, E	M, E			
В	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	SE	M, E	M, E			
С	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	NA	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) Al ₆ O ₃ N ₄ (12H)	γ-AION Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) Al ₆ O ₃ N ₄ (12H)	M, E			
D	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) M, E	M, E	M, E			
Е	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R) γ-AlON	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R) y-AlON	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R) γ-AlON	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R)	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R)	Al ₉ O ₃ N ₇ (27R) Al ₇ O ₃ N ₅ (21R)			
F	AlN γ-AlON	NA	Al ₉ O ₃ N ₇ (27R) AlN y-AlON	NA	AlN Al ₉ O ₃ N ₇ (27R) γ-AlON	AlN Al ₉ O ₃ N ₇ (27R)			
G	γ-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	y-AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R)	<i>y-</i> AlON Al ₃ O ₃ N Al ₉ O ₃ N ₇ (27R) Flaky	M, E	M, E			

NA = Not Analyzed

M = Molten

E = Evaporated SE = Slight Evaporation